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REMARKS

The Office has maintained the rejections made in the previous action. Claims 20 and 24 remain rejected under 35 U.S.C. 103(a) as being unpatentable over Fusegawa et al. (US 2003/0106484) (hereinafter: "Fusegawa") in view of JP 2003-297840 (hereinafter "JP '840"). Claims 21, 22, 25, 26, 28-30, 32-34, 36-38 and 40-45 remain rejected under 35 U.S.C. 103(a) as being unpatentable over Fusegawa and JP '840 and further in view of various combinations of Haas et al. (US 4,119,441) (hereinafter: "Haas"), Asayama et al. (U.S. Patent No. 6,641,888) (hereinafter: "Asayama") and Momoi et al. (U.S. Patent No. 2002/0024152) (hereinafter: "Momoi").

The Office identifies Fusegawa as disclosing a silicon wafer manufactured from a silicon single crystal having high interstitial oxygen concentration as recited in claims 20 and 24. JP '840 is cited as disclosing a relational expression between an interstitial oxygen concentration and heat treatment temperature as recited in claims 20 and 24. The remaining references are cited as disclosing various of the limitations of the remaining claims.

In response to the arguments traversing these rejections in the response filed October 25, 2007, to the first Office Action, the Office states:

> Applicants argue that neither of Fusegawa et al. (US 2003/0106484), JP (2003-297840), Haas et al (US

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> 4,119,441) Asayama et al (US 6,641,888) and Momoi et al (US 2002/0024152) teach or suggest the expression relating the interstitial oxygen concentration and temperature recited.in the instant application.
>
> This is not found persuasive because applicants attention is drawn to the point that non [sic, none] of the cited references ... has not been used alone, but combination rejection made over Fusegawa et al. (US 2003/0106484), JP (2003-297840) clearly teaches a manufacturing method of a silicon wafer, in which a silicon wafer that has been sliced from a silicon single crystal is heat treated in an oxidizing atmosphere wherein there is a relationship between oxygen concentration and heat treatment temperature (T) as recited in the previous office action.

The Office's characterization of applicants' arguments made in the response to the first action is not correct. Applicants did not separately argue the references. Applicants argued in the first response that the proposed <u>combination</u> of Fusegawa with JP '840 and/or with the other references cited in the Action, will not result in the method of the present invention as recited in the rejected claims. This argument is explained in further detail below.

Initially, however, applicants submit that the Office has not provided a proper rationale for combining Fusegawa with JP '840 as proposed in the final rejection. As explained in MPEP §2141 (III) (October, 2005):

> The key to supporting any rejection under 35 U.S.C. 103 is the clear articulation of the reason(s) why the claimed invention would have been obvious. The Supreme Court in *KSR* noted that the analysis supporting a

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rejection under 35 U.S.C. 103 should be made explicit. The Court quoting *In re Kahn*, 441 F.3d 977, 988, 78 USPQ2d 1329, 1336 (Fed. Cir. 2006), stated that "'[R]ejections on obviousness cannot be sustained by mere conclusory statements; instead, there must be some articulated reasoning with some rational underpinning to support the legal conclusion of obviousness.'" *KSR*, 550 U.S. at ___, 82 USPQ2d at 1396.

In the present rejections, the Office has not "articulated reasoning with some rational underpinning to support the legal conclusion of obviousness." Instead, the Office simply identifies JP '840 as "analogous art" (Action mailed July 26, 2007, page 3, line 15) and concludes that it would have been obvious "to increase confidence level in overall process control for manufacturing method of silicon wafer of Fusegawa ... in order to measure the strength of silicon wafer since oxygen concentration is effective for enhancing the strength of a silicon wafer, as suggested by JP (2003-297840)." However, the Office has not provided "some rational underpinning" for this conclusion or otherwise explained <u>why</u> a person of ordinary skill in the art would have been motivated to enhance the strength of a silicon wafer <u>in order to measure the strength of the wafer</u>.

For this reason alone, the final rejections are not proper and should be removed.

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Moreover, as has been previously explained, the combinations of prior art proposed by the Office will not result in the method of the present invention.

Fusegawa teaches the Czochralski (CZ) method [crystal pulling method], and discloses a method for making the interstitial oxygen concentration in a single silicon crystal low by controlling the pulling conditions (pulling speed, strength of magnetic field, rotary speed of crucible, and the like) and manufacturing high quality wafers. Furthermore, Fusegawa discloses heat treatment of this wafer for 100 minutes at 1150°C in a wet oxidizing atmosphere to examine the distribution of OSF occurrences in a wafer grown and manufactured under the conditions prescribed above. In addition, Fusegawa discloses that, from the standpoint of warp and slippage, the preferable oxygen concentration for the wafer described above is $6 - 10 \times 10^{17}$ atom/cm^3.

However, Fusegawa discloses nothing concerning the relationship between the interstitial oxygen concentration of the wafer and the heat treatment temperature of the wafer. Fusegawa discloses nothing more than carrying out pulling from a silicon melt with the oxygen concentration described above at a prescribed speed and prescribed temperature conditions, making BMD uniform and

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preparing a single crystal that does not have OSF and other defects.

In the JP '840 reference, heat treating a silicon wafer at a temperature that satisfies the following expression is disclosed.

$Oi < Oieq \cdot T exp(2\sigma SiO_2 \Omega / \gamma \kappa T)$.

The expression disclosed in JP '840 includes the thermal equilibrium concentration (solid solubility) [Oi]eq of the interstitial oxygen, and in paragraph [0022] of that publication, it is stated that "[Oi]eq (T) is the edge solubility (Randloeslichkeit) of the oxygen in the silicon at a given temperature T. Functions of this sort are discussed in Hull, R. (Ed.), "Properties of Crystalline Silicon", The Institution of Electrical Engineers, London, 1999, pp. 498-491.[1]

However, in "Properties of Crystalline Silicon" on p. 489, discloses that:

$[Oi]_{eq} = C_s \exp(-Es(K)/T)$ cm^{-3}

Cs and Es are in TABLE 1 on p. 490.

Cs and Es differ according to the reporter, and 11 combinations are given in TABLE 1 on p. 490.

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[1] A copy of "Properties of Crystalline Silicon" was submitted to the Office with the response filed October 25, 2007. For Office convenience, a copy of the document is attached hereto.

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In other words, in JP '840, the range of [Oi] is not clear and is not definite. Thus, in JP '840, nothing is written about the following specific expression required in the method of the present invention.

Oxygen concentration $\leq 2.123 \times 10^{21} \exp(-1.035/k(T + 273)$

The invention of the present application is established based on the expression described above regarding the relationship between the oxygen concentration of a silicon wafer without COP and the heat treatment temperature of the silicon wafer in the manufacturing of a silicon wafer.

The equation required in the method according to the invention in the present application cannot easily be conceived of from the equation in JP '840. Specifically, in terms of the oxygen concentration and the heat treatment temperature, the relational expression required according to the method of the present invention cannot be derived using the disclosure in the JP '840 reference, which shows an uncertain fixed relational expression.

Therefore, none of the combinations proposed by the Office will result in the method of the present application.

Removal of the 35 U.S.C. 013(a) rejections of the claims is believed to be in order and is respectfully requested.

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The foregoing is believed to be a complete and proper response to the Office Action dated January 14, 2008, and is believed to place this application in condition for allowance. If, however, minor issues remain that can be resolved by means of a telephone interview, the Examiner is respectfully requested to contact the undersigned attorney at the telephone number indicated below.

In the event that this paper is not considered to be timely filed, applicants hereby petition for an appropriate extension of time. The fee for any such extension may be charged to our Deposit Account No. 111833.

In the event any additional fees are required, please also charge our Deposit Account No. 111833.

Respectfully submitted,

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Attachment: "Properties of Crystalline Silicon"

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9.3 Solubility of O in c-Si

B. Pajot

April 1997

A INTRODUCTION

In the Czochralski (CZ) growth, O is introduced in molten silicon from the dissociation of silica from the crucible. It can also be introduced during float-zoned (FZ) growth when O-bearing gases are used, or by solid state diffusion.

The measurement of a solubility is that of a maximum concentration of isolated O in liquid or solid silicon under equilibrium with silicon oxides at a given temperature. Direct measurements of the O concentration in molten silicon contained in a silica crucible have recently been made using an electrochemical O sensor [1,2]. Common methods used to derive an O concentration are applicable only for the solid state. They are:

- Charged particle activation analysis (CPAA)
- Secondary ion mass spectroscopy (SIMS)
- Gas fusion analysis (GFA)
- Infrared (IR) absorption

CPAA, SIMS and GFA allow the determination of the overall O concentration. IR absorption is used to measure the concentration of interstitial oxygen atoms bonded to two nearest neighbour Si atoms (O_i). The IR method is based on the fact that the normal cooling rate of CZ silicon is too fast for noticeable precipitation of silicon oxides to occur so that metastable O_i concentrations comparable to the ones near the melting point (1413°C) are measured. The O_i concentration is determined from the room temperature absorption coefficient of an O_i vibration-rotation band at 9.04 μm using a suitable calibration factor. Within ±3%, there is now a world-wide agreement on a value of 3.1×10^{17} cm^{-2} for this factor [3]. Silica precipitates may be present in the crystal for very high oxygen concentrations or after some annealing treatments. They can alter the measurement as they show absorption in the same spectral region as the O_i band. An alternative in that case is to measure the absorption near liquid helium temperature (LHeT) [4]. Another one is to measure at room temperature a weak absorption band of O_i at 5.81 μm or 1720 cm^{-1} in wavenumber, which is free of interference with the absorption of the precipitates [5]. Another method for O_i determination is to measure, by an X-ray technique, the average lattice expansion due to the presence of O_i in the crystal [6]. The lattice expansion coefficient β can be expressed in terms of the increase Δa_0 of the lattice constant due to an increase ΔN of the concentration of a foreign atom as $\Delta a_0/(a_0\Delta N)$. For O_i, a reliable value of β is $(4.4 \pm 0.2) \times 10^{-24}$ cm^3 [7].

B LIQUID SOLUBILITY

A determination of the liquid solubility from thermodynamic calculations has been made by Carlberg [8]. It is based on an experimental value of the standard free-energy of solution of oxygen in liquid silicon in equilibrium with SiO_2. The solubility is thermally activated and can be expressed as $[O_{eq}]_{liq} = 1.3 \times 10^{24} \exp(-22370(K)/T)$ cm^{-3}. The value at the melting point is found to be 2.1×10^{18} cm^{-3} and the activation energy 2.0 eV. Ekhult and Carlberg [9] have also shown the importance of the Si/SiO equilibrium in melts with a lower O content. Comparable results have been obtained experimentally by GFA of quenched melts contained in a silica tube by Hirata and

Hoshikawa [10], who derived $[O_{eq}]_{liq} = 4.0 \times 10^{23} \exp(-2.0 \times 10^4(K)/T)$ cm^{-3}. Huang et al [11] used GFA and SIMS to evaluate the O concentration in samples very similar to those of [8]. The main difference in their results is a much weaker temperature dependence of the solubility (if any). A value of the solubility in the melt near the melting point between 2 and 3×10^{18} cm^{-3} seems a reasonable estimation.

The absolute values of the O concentrations obtained by direct measurements in a melt contained in a regular CZ crucible with an electrochemical O sensor [1] are larger than expected by about a factor of three. These values are very probably related to the method used, but the method gives significant evidence of an O concentration gradient related to the rotation of the crucible and to the O source (the concentration is larger near to the walls of the crucible than near to the centre). The existence of an O concentration gradient due to the rotation of the crucible is confirmed by IR determination of the radial distribution of O in different frozen melts by Togawa et al [12].

C SOLID SOLUBILITY

As-grown or as-quenched CZ silicon is oversaturated with O_i at room temperature and the concentration measured is a good evaluation of the concentration near to the melting point. A reasonable order of magnitude of the solid solubility at the melting point can already be derived from the results of the quenched melt experiments in sealed quartz tubes indicating O_i concentrations in the vicinity of 2×10^{18} cm^{-3}. This figure has also been obtained from the construction of the Si-O phase diagram in the Si-rich region by Carlberg [8]. The O_i concentration in CZ materials depends strongly on the growth parameters and it is generally less than the equilibrium solubility at the melting point. Precise adjustment of the growth parameters allows production of CZ silicon crystals with fairly uniform and controlled O concentrations. A review by Lin [13] on the incorporation of O shows that if usual growth parameters allow production of O_i concentrations between about 5 and 12×10^{17} cm^{-3}, O_i concentrations near to the solubility limit have been found in crystals grown with large crucible rotation rates (~30 rpm) or by applying a vertical magnetic field to the melt. The solubility is expected to decrease with temperature. Its measurement requires a physical situation where thermal equilibrium is reached at a given temperature. This has been typically done by annealing a CZ material at a given temperature to produce internal precipitation of silicon oxides or by making O diffuse at a given temperature in silicon with an initial low O_i content, e.g. FZ silicon. The thermally activated solubility is expressed as:

$$[O_{eq}]_{sol} = C_\infty \exp(-E_s(K)/T) \text{ cm}^{-3} \quad (1)$$

where C_∞ is a constant related to the entropy of solution and E_s the heat of solid solution in Kelvin (E_s(eV) = 8.617×10^{-5} E_s(K)). A summary of the results is presented in TABLE 1.

Part of the spreading of the experimental results has been analysed by Wilkes [22] as due to a combination of different factors including:

1. The difficulty in discriminating between the IR signatures of O_i and of the precipitates.
2. Contamination of the samples.
3. Measurements performed while equilibrium was not reached.
4. Different IR calibration factors.

In 1986, Mikkelsen [23] proposed a composite solubility curve yielding a heat of solution of 1.52 eV by bringing together experimental data from different origins. It corresponds to:

$$[O_{eq}]_{sol} = 9.0 \times 10^{22} \exp(-1.76 \times 10^4(K)/T) \text{ cm}^{-3} \quad (2)$$

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To obtain this expression, the data using IR measurements were replotted using a common IR calibration factor (3.03×10^{17} cm^{-2}) very close to the one recommended above. Thus, EQN (2) is recommended for the solubility of O in silicon, with a domain of validity between the silicon melting point and about 850°C. For these limits, the solubilities derived from EQN (2) are 2.58×10^{18} and 1.36×10^{16} cm^{-3}, respectively. Between 850 and 700°C, the apparent solubility versus T^{-1} shows a flattening and Newman [24] even reports an increase of the solubility below 700°C to about 10^{17} cm^{-3} near 500°C. This is due to the combined increase of the concentration of oxide precipitates and to a decrease of their sizes and this must be considered as a pseudo-equilibrium situation.

TABLE 1 Summary of data relating to O solid solubility. E_s should be multiplied by 8.617×10^{-5} to read in eV.

E_s 10^4 (K)	C_s 10^{22} (cm^{-3})	Temp. range (°C)	Method	Detection	Ref
1.09	0.12	1000–1250	O precipitation	IR (LHeT)	[14]
2.67	18	1250–1400	Thermal donors	Spreading resistance	[15]
1.82	12	1100–1200	O precipitation	IR (77 K)	[16]
1.39	0.82	-	O diffusion	X-rays	[6]
1.15	0.19	780–1100	O precipitation	IR (77 and 300 K)	[17]
1.24	0.28	1000–1280	O diffusion	CPAA	[18]
1.20	0.2		O precipitation in neutron-irradiated silicon	IR (300 K)	[19]
1.18	0.064	600–1240	O diffusion	SIMS and IR (300 K)	[20]
1.39	0.71	650–1050	O precipitation	IR (LHeT and 300 K)	[4]
1.40	0.95	1000–1375	O diffusion	CPAA	[21]
2.54	740	near 1413	O-Si phase diagram	-	[8]
1.62	2.6	850–1200	Composite from [4], [16] and [20]	-	[4]

A decrease of the O solubility in n^+ Sb-doped CZ silicon has been reported [25]. Possible reasons are: 1) a reduction of the O solubility in the melt due to antimony, 2) an increase of the O evaporation through Sb-containing oxides and 3) a decrease of the segregation of O. The measurements by Huang et al [11] of the O concentration in frozen melts containing increasing Sb concentrations show that for [Sb] < 1% at, the O solubility is unchanged, but when [Sb] increases, the O solubility increases to reach about 10^{19} cm^{-3} for [Sb] near 2% at. These results seem to rule out the first assumption.

D CONCLUSION

A composite value of the O solubility in solid silicon $[O_{eq}]_{sol}$ at the melting point is 2.6×10^{18} cm^{-3}, but in most CZ crystals, the maximum $[O_i]$ measured in as-grown CZ silicon is $1.2–1.4 \times 10^{18}$ cm^{-3} with a small concentration of precipitated [O] and TDs. Values near or slightly above the solubility limit have been found only in some 'magnetic' crystals [13], and this means that for most other CZ materials, the solubility limit is not reached. The above value of $[O_{eq}]_{sol}$ agrees acceptably with the value of 2.0×10^{18} cm^{-3} obtained from a determination of the Si/SiO_2 phase diagram.

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